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June 21, 2001

BOX PCT

Commissioner for Patents
 Washington, D.C. 20231

PCT/JP99/07006 ✓
 -filed December 14, 1999 ✓



Re: Application of Masayuki YAMANA, Tsukasa AGA, Masahiro MIYAHARA, ✓
 Masaki FUKUMORI and Ryosuke HARA ✓
 AQUEOUS WATER- AND OIL-REPELLENT DISPERSION
 Assignee: **DAIKIN INDUSTRIES, LTD.**
 Our Ref: Q64814

Dear Sir:

The following documents and fees are submitted herewith in connection with the above application for the purpose of entering the National stage under 35 U.S.C. § 371 and in accordance with Chapter II of the Patent Cooperation Treaty:

- ☒ an English translation of the International Application.
- ☒ a Notification Concerning Submission or Transmittal of Priority Document.
- ☒ an English translation of Article 34 amendments (annexes to the IPER).
- ☒ an Information Disclosure Statement and Form PTO-1449 listing the ISR references.

The Declaration and Power of Attorney and Assignment will be submitted at a later date.

It is assumed that copies of the International Application, the International Search Report, the International Preliminary Examination Report, and any Articles 19 and 34 amendments as required by § 371(c) will be supplied directly by the International Bureau, but if further copies are needed, the undersigned can easily provide them upon request.

Assignment for published patent application is: **DAIKIN INDUSTRIES, LTD..**

The Government filing fee, after entry of the Article 34 Amendment, is calculated as follows:

Total claims	3 - 20 =	x \$18.00 =	\$0.00
Independent claims	1 - 3 =	x \$80.00 =	\$0.00
Base Fee			\$860.00

TOTAL FEE \$860.00

A check for the statutory filing fee of \$860.00 is attached. You are also directed and authorized to charge or credit any difference or overpayment to Deposit Account No. 19-4880. The Commissioner is hereby authorized to charge any fees under 37 C.F.R. §§ 1.16, 1.17 and



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1.492 which may be required during the entire pendency of the application to Deposit Account No. 19-4880. A duplicate copy of this transmittal letter is attached.

Priority is claimed from December 22, 1998 based on Japanese Application No. 364298/1998.

Respectfully submitted,

Abraham J. Rosner
Registration No. 33,276

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Date: June 21, 2001

DESCRIPTION

AQUEOUS WATER- AND OIL-REPELLENT DISPERSION

5 FIELD OF THE INVENTION

The present invention relates to an aqueous water- and oil-repellent dispersion comprising a polymer containing a polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group, which can impart the water- and oil repellency to various fibers.

RELATED ARTS

It is well known that a polymer of a polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group can be used as a water- and oil-repellent for a fibrous fabric. An aqueous dispersion which is prepared by dispersing the polymer in an aqueous medium by means of an emulsifier widely has the industrial utilization. However, the water- and oil-repellency of a fibrous fabric treated with usual conventional aqueous dispersions is not satisfactory for resistance, namely durability, to physical action such as friction. In addition, the conventional aqueous dispersions do not have excellent storage stability even if

they have the durable water- and oil repellency.

SUMMARY OF THE INVENTION

One of the objects of the present invention is to
5 provide an aqueous water- and oil-repellent dispersion
having durable water- and oil-repellency and excellent
storage stability.

The present invention provides an aqueous water- and
oil-repellent dispersion comprising:

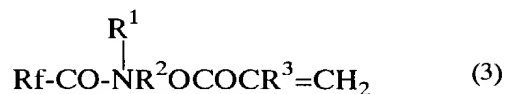
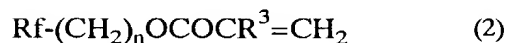
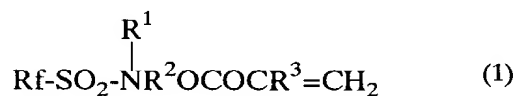
- 10 (A) a homopolymer or copolymer comprising at least one
polymerizable compound having a perfluoroalkyl or
perfluoroalkenyl group and an acrylate or methacrylate
group, or
a copolymer comprising said polymerizable compound and
15 another compound copolymerizable therewith,
(B) an organic solvent which is tripropylene glycol, and
(C) a surfactant.

DETAILED EXPLANATION OF THE INVENTION

20 In the copolymer, which is the polymer (A), comprising
the polymerizable compound having the perfluoroalkyl or
perfluoroalkenyl group and the acrylate or methacrylate
group and the another compound copolymerizable therewith,
the former compound is in the amount of at least 25 % by
25 weight, preferably at least 40 % by weight based on the

weight of the copolymer.

Examples of the polymerizable compound having the perfluoroalkyl or perfluoroalkenyl group and the acrylate or methacrylate group include (meth)acrylate esters
5 represented by the formulas:



wherein Rf is a perfluoroalkyl or perfluoroalkenyl group having 3 to 21 carbon atoms,

10 R¹ is a hydrogen atom or an alkyl group having 1 to 10 carbon atoms,

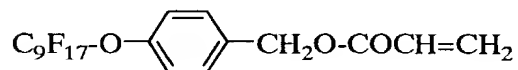
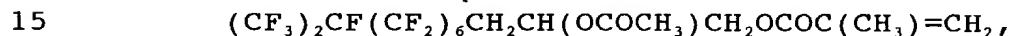
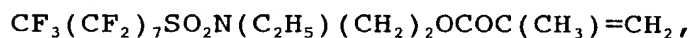
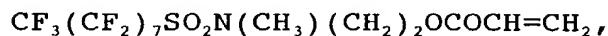
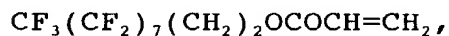
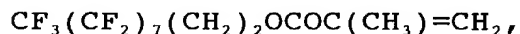
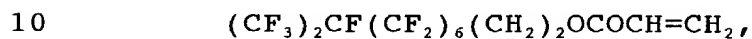
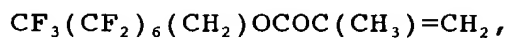
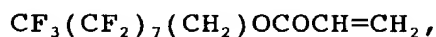
R^2 is an alkylene group having 1 to 10 carbon atoms,

R^3 is a hydrogen atom or a methyl group,

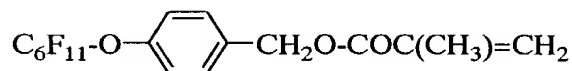
Ar is an aryl group which optionally has a substituent group, and

5 n is an integer of 1 to 10.

Specific examples of the polymerizable compound include:



and



The another copolymerizable compound are various.

Examples of the another copolymerizable compound include:

20 (1) acrylic acid and methacrylic acid, and methyl, ethyl, butyl, isobutyl, t-butyl, propyl, 2-ethylhexyl, hexyl,

decyl, lauryl, stearyl, isobornyl, β -hydroxyethyl, glycidyl, phenyl, benzyl and 4-cyanophenyl esters thereof; (2) vinyl esters of fatty acids such as acetic acid, propionic acid, caprylic acid, lauric acid and stearic acid; (3) styrene compounds such as styrene, α -methylstyrene and p-methylstyrene; (4) vinyl halides and vinylidene compounds such as vinyl fluoride, vinyl chloride, vinyl bromide, vinylidene fluoride and vinylidene chloride; (5) fatty acid allyl esters such as allyl heptanoate, allyl caprylate and allyl caproate; (6) vinyl alkyl ketones such as vinyl methyl ketone and vinyl ethyl ketone; (7) acryl amides such as N-methylacrylamide and N-methylolmethacrylamide; and (8) dienes such as 2,3-dichloro-1,3-butadiene and isoprene.

The organic solvent in the present invention is tripropylene glycol, $\text{H}[\text{OCH}_2\text{CH}(\text{CH}_3)]_3\text{OH}$.

The amount of the organic solvent (B) may be from 5 to 200 parts by weight, for example from 10 to 100 parts by weight, particularly from 20 to 60 parts by weight, based on 100 parts of the polymer (A).

The surfactant (C) used for dispersing the polymer and the organic solvent may be a cationic emulsifier, an anionic emulsifier or a nonionic emulsifier. The surfactant is preferably the cationic emulsifier, the nonionic emulsifier or a mixture of both. In the case of the mixture, a preferable weight ratio of the cationic

emulsifier to the nonionic emulsifier is from 1:9 to 9:1. Specific examples of the cationic emulsifier include dodecyl trimethyl ammonium acetate, trimethyl tetradecyl ammonium chloride, hexadecyl trimethyl ammonium bromide, trimethyl octadecyl ammonium chloride, (dodecylmethylbenzyl) trimethyl ammonium chloride, benzyl dedecyl dimethyl ammonium chloride, methyl dodecyl di(hydropolyoxyethylene) ammonium chloride, benzyl dodecyl di(hydropolyoxyethylene) ammonium chloride, benzyl dodecyl di(hydropolyoxyethylene) ammonium chloride and N-[2-(diethylamino)ethyl]oleamide hydrochloride. Specific examples of the nonionic emulsifier include a condensation product of ethylene oxide with hexyl phenol, iso-octyl phenol, hexadecanol, oleic acid, alkane(C_{12} - C_{16})thiol, sorbitan monofatty acid (C_7 - C_{19}) or alkyl(C_{12} - C_{18})amine and the like.

The amount of the surfactant (C) may be from 0.01 to 30 parts by weight, for example from 1 to 20 parts by weight, based on 100 parts by weight of the polymer (A).

The dispersion according to the present invention can be prepared by emulsion-polymerizing the polymerizable compound(s) in water accompanied by the organic solvent (B) in the presence of the surfactant to give an emulsion of the polymer (A). Water and/or the surfactant may be added to the emulsion of the polymer (A).

Examples of a suitable substrate, to which the

dispersion according to the present invention is applied, include a film, a fiber, a yarn, a woven fabric, a carpet, a filament made from a natural polymer material, a modified natural polymer material and a synthetic polymer material, and a product made from a fiber and a yarn. The substrate is preferably a textile which is in the form of a fiber, a yarn or a fabric.

The dispersion according to the present invention can be applied to the substrate preferably by coating, dipping, spraying, padding, roll coating, or combination of these procedures. For example, a padding bath having the bath solid content of 0.1 to 10 % by weight can be used. The substrate is padded in the bath, and then excess liquid is usually removed by a squeezing roll to give the dry pickup amount (the weight of dry polymer on the substrate) of from 0.01 to 10 % by weight based on the weight of the substrate. Then, the treated substrate is preferably heated at 100-200 °C.

20 PREFERRED EMBODIMENTS OF THE INVENTION

Examples and Comparative Examples are shown hereinafter to illustrate the present invention in detail.

Properties are determined as follows:

Water- and oil-repellency

25 The polymer dispersion liquid is diluted with water to

give a treatment liquid having a solid content of 0.08 % by weight. A polyester fabric is immersed in the treatment liquid, squeezed with a mangle to give a wet pickup of 65%, dried at 100°C for two minutes, heated at 160°C for one hour, and then subjected to an evaluation of water- and oil-repellency.

The water-repellency is expressed by the water repellency No. (cf. the following Table 1) determined by the spray method according to JIS (Japanese Industrial Standard) L-1092.

The oil-repellency is determined by dropping several drops of a test solution shown in AATCC-TM118 (Table 2) on two positions of a surface of a test cloth and observing the penetration state of the drops after 30 seconds. The maximum point at which the test solution exhibits no penetration is expressed by the oil-repellency.

Table 1

Water repellency No.	State
5	No wet on the surface
4	Slight wet on the surface
3	Partial wet on the surface
2	Wet on the surface
1	Wet over the whole surface

Table 2

Oil repel- lency	Test solution	Surface tension (dyne/cm, 25°C)
8	n-Heptane	20.0
7	n-Octane	21.8
6	n-Decane	23.5
5	n-Dodecane	25.0
4	n-Tetradecane	26.7
3	n-Hexadecane	27.3
2	n-Hexadecane/Nujol mixture solution (35/65 by weight)	29.6
1	Nujol	31.2
0	Inferior to 1	-

Storage stability

After the aqueous dispersion (solid content: 30 % by weight) is stored at 40°C for one month, the generation of precipitation is observed.

○: No precipitation

△: Slight precipitation

×: Much precipitation

10

Example 1

100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 50 g of stearyl acrylate, 2 g of N-methylol acrylamide, 200 g of pure water, 40 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkylphenyl ether were charged into a 500 mL

flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutylamidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Example 2

100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 50 g of stearyl acrylate, 2 g of 3-chloro-2-hydroxypropyl methacrylate, 200 g of pure water, 30 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkylphenyl ether were charged into a 500 mL flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutylamidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Example 3

100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds

wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 25 g of stearyl acrylate, 25 g of 2-ethylhexyl methacrylate, 200 g of pure water, 80 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkyl ether were charged into a 500 mL flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutylamidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Example 4

15 100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 50 g of stearyl acrylate, 2 g of diacetone acrylamide, 200 g of pure water, 50 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl trimethyl ammonium chloride and 10 g of polyoxyethylenealkylphenyl ether were charged into a 500 mL flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutylamidine dihydrochloride was added and the reaction was continued for 5 hours to give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Example 5

5 100 g of $C_nF_{2n+1}CH_2CH_2OCOCH=CH_2$ (a mixture of compounds wherein n is 6, 8, 10, 12 and 14 (average of n is 8)), 25 g of stearyl acrylate, 25 g of 2-ethylhexyl methacrylate, 2 g of N-methylolacrylamide, 200 g of pure water, 60 g of tripropylene glycol, 0.3 g of acetic acid, 4 g of alkyl
10 trimethyl ammonium chloride and 10 g of polyoxyethylene-alkyl ether were charged into a 500 mL flask and emulsified at 60°C for 15 minutes by means of ultrasonic wave with stirring. 0.75 g of azobisisobutyramidine dihydrochloride was added and the reaction was continued for 5 hours to
15 give an aqueous dispersion of a polymer.

The water- and oil-repellency and the storage stability were evaluated. The results are shown in Table 3.

Comparative Example 1

20 The procedure of Example 1 was repeated except that 40 g of propylene glycol was used instead of 40 g of tripropylene glycol.

Comparative Example 2

25 The procedure of Example 1 was repeated except that 40

g of dipropylene glycol was used instead of 40 g of tripropylene glycol.

Comparative Example 3

5 The procedure of Example 2 was repeated except that 30 g of propylene glycol was used instead of 30 g of tripropylene glycol.

Comparative Example 4

10 The procedure of Example 2 was repeated except that 30 g of dipropylene glycol was used instead of 30 g of tripropylene glycol.

Comparative Example 5

15 The procedure of Example 4 was repeated except that 50 g of propylene glycol was used instead of 50 g of tripropylene glycol.

Tripropylene glycol used in the present invention is excellent in no harm. The aqueous dispersion according to the present invention is effective for decreasing the environmental pollution and has durable water- and oil-repellency and excellent storage stability.

CLAIMS

1. (amended) An aqueous water- and oil-repellent dispersion comprising:

5 (A) a homopolymer or copolymer comprising at least one polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group, or

a copolymer comprising said polymerizable compound and
10 another compound copolymerizable therewith,

(B) an organic solvent which is tripropylene glycol, and

(C) a surfactant,

wherein the amount of the organic solvent (B) is from 5 to 200 parts by weight, based on 100 parts by weight of the
15 polymer (A), and the polymer (A) is emulsion-polymerized in water accompanied by the organic solvent (B).

2. (deleted)

20 3. The dispersion according to claim 1, wherein the amount of the organic solvent (B) is from 10 to 100 parts by weight, based on 100 parts by weight of the polymer (A).

4. A textile, to which the dispersion according to claim 1
25 is applied.

ABSTRACT

An aqueous water- and oil-repellent dispersion containing: (A) a homopolymer or copolymer containing at least one polymerizable compound having a perfluoroalkyl or perfluoroalkenyl group and an acrylate or methacrylate group, or a copolymer containing said polymerizable compound and another compound copolymerizable therewith, (B) an organic solvent which is tripropylene glycol, and (C) a surfactant, has durable water- and oil-repellency and excellent storage stability.

Declaration and Power of Attorney for Patent Application

特許出願宣言書及び委任状

Japanese Language Declaration

日本語宣言書

下記の氏名の発明者として、私は以下の通り宣言します。

私の住所、私書箱、国籍は下記の私の氏名の後に記載された通りです。

下記の名称の発明に関して請求範囲に記載され、特許出願している発明内容について、私が最初かつ唯一の発明者（下記の氏名が一つの場合）もしくは最初かつ共同発明者であると（下記の名称が複数の場合）信じています。

上記発明の明細書（下記の欄でX印がついていない場合は、本書に添付）は、

☐ ____月 ____日に提出され、米国出願番号または特許協定条約

国際出願番号を _____ とし、

（該当する場合） _____ に訂正されました。

私は、特許請求範囲を含む上記訂正後の明細書を検討し、内容を理解していることをここに表明します。

私は、連邦規則法典第37編第1条56項に定義されるとおり、特許資格の有無について重要な情報を開示する義務があることを認めます。

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

AQUEOUS WATER- AND OIL-REPELLENT

DISPERSION ✓

the specification of which is attached hereto unless the following box is checked:

☒ was filed on December 14, 1999 ✓
as ~~United States Application Number~~ XXXX
PCT International Application Number

PCT/JP99/07006 ✓ and was amended on

December 21, 2000 (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56.

Japanese Language Declaration

(日本語宣言書)

私は、米国法典第35編第119条(a)-(d)項又は第365条(b)項に基づき下記の、米国以外の国の少なくとも一カ国を指定している特許協力条約第365条(a)項に基づく国際出願、又は外国での特許出願もしくは発明者証の出願についての外国優先権をここに主張するとともに、優先権を主張している本出願の前に出願された特許または発明者証の外国出願を以下に、枠内をマークすることで、示しています。

Prior Foreign Applications

外国での先行出願

364298/1998✓ (Number) (番号)	Japan✓ (Country) (国名)
_____ (Number) (番号)	_____ (Country) (国名)
_____ (Number) (番号)	_____ (Country) (国名)

私は、第35編米国法典119条(e)項に基づいて下記の米国特許出願規定に記載された権利をここに主張致します。

_____ (Application No.) (出願番号)	_____ (Filing Date) (出願日)
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私は、下記の米国法典第35編第120条に基づいて下記の米国特許出願に記載された権利、又は米国を指定している特許協力条約第365条(c)に基づく権利をここに主張します。又、本出願の各請求範囲の内容が米国法典第35編第112条第1項又は特許協力条約で規定された方法で先行する米国特許出願に開示されていない限り、その先行米国出願書提出日以降で本出願書の日本国内又は特許協力条約国際出願提出日までの期間中に入手された、連邦規則法典第37編第1条第56項で定義された特許資格の有無に関する重要な情報について開示義務があることを認識しています。

_____ (Application No.) (出願番号)	_____ (Filing Date) (出願日)
_____ (Application No.) (出願番号)	_____ (Filing Date) (出願日)

私は、私自身の知識に基づいて本宣言中で私が行う表明が真実であり、かつ私の入手した情報と私の信ずるところに基づく表明が全て真実であると信じていること、さらに故意になされた虚偽の表明及びそれと同等の行為は米国法典第18編第1001条に基づき、罰金または拘禁、もしくはその両方により処罰されること、そしてそのような故意による虚偽の声明を行えば、出願した、又は既に許可された特許の有効性が失われることを認識し、よってここに上記のごとく宣誓を致します。

I hereby claim foreign priority under Title 35, United States Code, Section 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Priority Not Claimed

優先権主張なし

22/December/1998✓ (Day/Month/Year Filed) (出願年月日)	<input type="checkbox"/>
_____ (Day/Month/Year Filed) (出願年月日)	<input type="checkbox"/>
_____ (Day/Month/Year Filed) (出願年月日)	<input type="checkbox"/>

I hereby claim the benefit under Title 35, United States Code, Section 119(e) of any United States provisional application(s) listed below.

_____ (Application No.) (出願番号)	_____ (Filing Date) (出願日)
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I hereby claim the benefit of Title 35, United States Code Section 120 of any United States application(s), or 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code Section 112, I acknowledge the duty to disclose any material information which is material to patentability as defined in Title 37, Code of Federal Regulations, Section 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application:

_____ (Status: Patented, Pending, Abandoned) (現況: 特許許可済、係属中、放棄済)
_____ (Status: Patented, Pending, Abandoned) (現況: 特許許可済、係属中、放棄済)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

(日本語宣言書)

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith (*list name and registration number*)

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唯一又は第一発明者名		Full name of sole or first inventor <u>Masayuki YAMANA</u>	
発明者の署名	日付	Inventor's signature <u>Masayuki Yamana</u>	Date <u>May 22, 2001</u>
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第二共同発明者名(該当する場合)		Full name of second joint inventor, if any <u>Tsukasa AGA</u>	
第二発明者の署名	日付	Second inventor's signature <u>Tsukasa AGA</u>	Date <u>may 22, 2001</u>
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(第三以降の共同発明者についても同様に記載し、署名をするこ (Supply similar information and signature for third and subsequent joint inventors.)

Japanese Language Declaration (日本語宣言書)

300 第三共同発明者名 (該当する場合)		Full name of third joint inventor, if any <u>Masahiro MIYAHARA</u>	
第三発明者の署名	日付	Third inventor's signature <u>Masahiro MIYAHARA</u>	Date <u>May 22, 2001</u>
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400 第四共同発明者名 (該当する場合)		Full name of fourth joint inventor, if any <u>Masaki FUKUMORI</u>	
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第六共同発明者名 (該当する場合)		Full name of sixth joint inventor, if any	
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郵便の宛先		Post office address	